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DISSEMINATED BY-

ARMED SERVICES EXPLOSIVES SAFETY BOARD

WASHINGTON, D.C. 20315

JULY 1968

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FOREWORD

The abstracts contained herein were disseminated between July 1967 and June 1968.

The "Explosives Accident/Incident Dissemination Program" was instituted in 1961 by the Armed Services Explosives Safety Board and is participated in on a voluntary basis by private industry and Government Agencies.

This program is intended to prevent, by means of expeditious dissemination of information, the reoccurrence of explosives incidents.

B. B. Abrams

B. B. ABRAMS
Colonel, USA
Chairman

I N D E X

ARMED SERVICES EXPLOSIVES SAFETY BOARD
Washington, D. C. 20315

EXPLOSIVES INCIDENT REPORT NO. 220

Propellant Mix House Fire

Description: A fire occurred in a mixer containing a 720 lb charge of a double base mix. The operations had been running normal during the entire mix cycle and the mixer had entered the final cool down period just prior to pulling the mixer. At the time of the incident the propellant temperature was 108°F. The normal discharge temperature of the mix ranges from 95°F to 110°F. The operator was remotely monitoring the operations via television and a sound transmitter system. He heard the mixer lid jump up and slam down and then he saw smoke coming from the mixer. At this point he shutdown the mixer and then observed that the automatic sprinkler system had tripped. Smoke was then observed coming out of the tunnels leading into the building and the fire continued until basically all the propellant mix in the mixer was consumed.

There was no injury to personnel.

The total damage included:

a. 720 lbs of propellant in the mixer valued at $936 and 880 lbs. of slurried Ammonium Perchlorate which was contaminated by water and valued at $300. The total material loss thus was $1,236.

b. The total damage to the mixer and the building has been estimated at $4,500. The interior of the mixer was scorched. The side walls of the mixer and the blades were slightly warped which will result in a complete overhaul of the mixer.

c. The mixer cover contained burn out panels made of 0.020" aluminum. These had been installed after a previous mixer fire. It was expected that these burn out panels would be destroyed immediately allowing water to enter the mixer in an attempt to limit the damage due to heat. This did not work and a study is now underway to determine what material can be used to give a satisfactory burn out panel. One panel of the mixer cover had a hole burned in it covering only about 5% of its surface area.

d. The teflon inserts in both journals were removed and inspected and it was found that no unusual burning on the insert surfaces had been experienced.

e. It was also determined that the electric grounds on the system were attached and were in good condition and the records indicate that the operators were following the Standard Operating Procedure.

f. The burned propellant remaining after the incident was examined and two pieces of magnetic metal were found. These pieces measured 2 3/4" and 1 1/2" long. They were about 1/4" wide and 1/16" thick.

The conclusions reached were that the fire was most probably caused by the two foreign objects being pinched between the mixer bowl and the blades.

Corrective Action:

a. At present the Nitrocellulose, Ammonimum Perchlorate, and Aluminum are either being screened or passed through a metal detector prior to their entry into the mixer. In the future the antioxidant will also be screened until a metal detector suitable for use on the material is properly installed. The manufacturer of the HMX assured that the material was checked for foreign metal objects prior to shipment. Prior to this incident an investigation was underway to determine a suitable metal detector to check this material and will be continued to try to obtain a suitable metal detector. Because of the nature and the type of the material it is virtually impossible to screen the material without presenting other problems that could even be more serious in nature.

b. The present burn out panels will be replaced. The most logical candidate is still aluminum panels with the thickness being reduced from 0.020" to 0.010".

c. Two systems are being investigated that will automatically open the lid when the temperature in the mixer increases above a certain level. One of the systems is an electrical system and the other system is based on a counter weight fusible link type arrangement.

Reference Number of this Report: EI-220

Duplication of this Report is authorized.

ARMED SERVICES EXPLOSIVES SAFETY BOARD
Washington, D. C. 20315

EXPLOSIVES INCIDENT REPORT NO. 221

Explosion in Research Laboratory

The following is an abstract of the incident report:

a. An explosion occurred in a laboratory of the main research building causing fire, a minor secondary explosion, noxious, irritating fumes and smoke, damage to the contents of the room and to the adjacent laboratory.

b. There were two chemists within the room at the time of the explosion. One of the chemists had been working on the synthesis and characterization of ethylene dioxyamineperchlorate (EDAP) a sensitive material. It is believed that the material was being purified and during some stage of this operation a low-order explosion occurred.

c. The explosion resulted in the death of the two individuals within the room. One individual died of shock due to blood loss and chemical-thermal burns. The cause of death of the second individual was cerebral edema, secondary to anoxemia, secondary to respiratory insufficiency, secondary to inhalation of noxious gases.

Reference Number of This Report: EI-221

Duplication of this Report is Authorized.

4/1/68

ARMED SERVICES EXPLOSIVES SAFETY BOARD
WASHINGTON, D. C. 20315

EXPLOSIVES INCIDENT REPORT NO. 222

Explosion in Ordnance Plant - Lead Azide Weighing Station

The following is an abstract of the incident report:

a. An explosion occurred at one of two lead azide weighing stations in a building of the Ordnance Plant, causing fatal injury of the worker who was weighing at this station. Thirteen other workers in the building suffered lost time injuries.

b. At the time of the accident, both Line 1 and Line 2 were in operation in the Main Mix Room. Each line consists of four Ordnance workers. Two operators work on one side of the table. The first operator weighs RDX and passes it to a second operator who transfers it to a flask containing Freon and Cabosil. The flasks are then passed across the table where a third operator weighs lead azide, and washes it into the flask using Freon. The fourth operator washes down the neck of the flask and caps it with plastic film and paper tape. A Quality Control Inspector is positioned behind each pair of Ordnance Workers. The explosion occurred at the lead azide weighing station, Line 1.

c. The fatally injured worker had obtained a bag of lead azide (approximately 15 pounds in a muslin cloth) from the "Wash Room" shortly prior to the accident. This bag of lead azide which had been washed with Freon, then immersed in Freon, and which was still Freon-wet, was the source of a detonation which caused the fatality and other injuries. It is believed that the fatally injured worker was beginning to "spread" the lead azide using a plastic spatula as he applied additional Freon from a spray nozzle. A minor detonation in the immediate work area may have initiated the bag of lead azide. However, there was no evidence that other material in the building detonated or burned. The stainless steel work table was blown apart at this weighing station. The floor beneath was damaged. Sheetrock sheathing inside the building was broken throughout the building. The frame exterior walls were moved outward but framing timber was not visibly damaged. The fatally injured Ordnance Worker was decapitated and his body was blown outside the building.

d. The evidence was not sufficient to establish a definite cause for the accident or to indicate a most probable cause. No significant departure from established practices could be identified. Possible causes include: (1) impact initiation by dropping Freon spray nozzle or scale pan; (2) electrostatic initiation from electric charge in Freon spray or on the person of the fatally injured employee; or (3) friction initiation from insertion of plastic spatula into lead azide. It is considered possible that a small amount of lead azide became dry at some undetermined location in the immediate work area; was initiated by impact, electrostatic discharge, or friction; and propagated to the Freon-wet lead azide.

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e. Corrective action taken: As no definite cause for the accident could be established, al? operations are being reviewed and modified where improved safety may be obtained.

Reference Number of this Report: ASESB 1218

Duplication of this Report is Authorized.

ARMED SERVICES EXPLOSIVES SAFETY BOARD
Nassif Building
Washington, D C 20315

OPERATIONAL INCIDENT REPORT NO. 107

Instrument Failure

Description: A flow transmitter in high-pressure hydrogen service had a failure in its internal mechanism resulting in rupture of disc provided to relieve excessive internal pressure in the transmitter.

Cause: A blow out plug provided to relieve pressure on the external housing after rupture of the internal disc failed to provide sufficient venting capacity, and the housing blew apart. Escaping hydrogen caught fire, the resulting flame being initially about 20 feet high and then subsiding to a steady flame about 6 feet long.

Action: Fire was extinguished in about 2-3 minutes and hydrogen source was shut off at about the same time. Hydrogen flow was resumed about 25 minutes later with instrument being by-passed.

There was no damage other than to the instrument (which the manufacturer has already replaced) no injuries, and no loss of process material.

The instrument was rated as suitable for this application, and the manufacturer has been given the instrument in question for study.

Preventive Measures: Engineering and instrumentation staff are studying the instrument in question and working closely with the manufacturer to determine cause of failure and prevent future failures Instrument was not supposed to fail under conditions being used. Instrument location was already safest possible (outside at moderate elevations).

(REPORTED BY MANUFACTURING CHEMISTS' ASSOCIATION, INC.)

Reference Number of this Report: OI-107

Duplication of this Report is authorized

7/5/67

ARMED SERVICES EXPLOSIVES SAFETY BOARD
Nassif Building
Washington, D. C. 20315

OPERATIONAL INCIDENT REPORT NO. 108

Explosive Accident in Dynamite Factory

Description:

On the 22nd February, 1967, at 10.28, a kneading house for dynamite exploded. Two workers in the house, 57 and 52 years of age, were killed. They had worked with the company for 38 and 34 years, respectively. Secondarily, about 3 minutes later a partial detonation occurred in an injector nitration plant about 80 metres from the kneading house. The nitration plant is automatic and was at the time unmanned. No personal injuries were caused in the plant outside the kneading house.

The kneading house was of timber and had a double Draiswerke kneading machine of an old revolving type. The kneading chamber was lined with copper. In the kneading house about 275 kilos Extra dynamite type IV, 140 kilos gelatined notroglycerine/nitroglycol and 120 kilos nitroglycerine 50/50 exploded. About 200 kilos notrotoluene mixture (N-content 11,8%) and about 30 kilos notrocellulose with 30% water did not take part in the explosion. The explosion took place just before lunch time. The third man of the work team had left for the workers mess. The investigation has clarified that the kneading machine was not in operation at the time of explosion. Probably, initiation was caused when ammonium nitrate was added into one of the mixing chambers. Ammonium nitrate is kept in cases of stainless steel and shall be discharged into the kneading chamber by two men. Parts of corpses, which have been found, indicate that only one worker was near the centre of the explosion, and probably, against the regulations, the filling of ammonium nitrate was made by only one worker.

By the ground vibration two control instruments in the nitration plant were damaged. The glycerine flow increased and thereby the nitration temperature rose above 60°C compared to normally 47°C. Registration of the nitration temperature is only possible up to 60°C. No impulse for restriction of the glyceri..e flow was released. The signal which breaks the nitration at 54° was also put out of operation. The faulty nitration continued for 3 minutes, and then an explosion started in the coil cooler just after the nitration injector and was interrupted in the next tubular cooler.

Preventive Measures:

In order to prevent a repetition the glycerine pipe has been provided with a diaphragm, which prevents overdosage of glycerine. A further contact breaker has been installed to break the operation at 54°C. A device for remote-interruption of the electric energy for the nitration has been arranged.

(Foreign source)

Reference Number of this Incident: OI-108

Duplication of this report is authorized.

8/1/67

ARMED SERVICES EXPLOSIVES SAFETY BOARD
Nassif Building
Washington, D. C. 20315

OPERATIONAL INCIDENT REPORT NO. 109

Pyrotechnic Composition Flash Fire

Description:

Pyrotechnic composition (Boron and Potassium Nitrate) was blended, screened and then placed into aluminum containers. The covered container was dropped by the operator and this resulted in a flash fire followed by explosions. The operator died as a result of severe burns and a nearby operator received minor abrasions and bruises.

Cause:

Ignition of pyrotechnic composition when container was dropped.

Action:

Line personnel were evacuated and a fire alarm was turned in after the first explosion. The fire propagated to adjacent material when a second explosion occurred. After it was determined that no other explosives were in the area, the fire department laid hose lines and the fire was extinguished.

Recommendations:

Employees handling sensitive pyrotechnic compositions should be instructed, trained and supervised in the specific hazards involved and the handling techniques to be followed.

Supervisors should maintain a continuous program of follow-up, reinstruction and enforcement of regulations with each employee.

Continuous cleaning, to prevent accumulation of dust, should be carried out as frequently as local circumstances require for maintaining safe conditions.

Process requirements should be reviewed to determine whether blending and screening operations could be conducted separately in order to reduce dust accumulation.

Reference Number of this Report: OI-109

Duplication of this report is authorized.

8/1/67

ARMED SERVICES EXPLOSIVES SAFETY BOARD
Nassif Building
Washington, D. C. 20315

OPERATIONAL INCIDENT REPORT NO. 110

Nitration Explosion

Description: An organic intermediate (to be nitrated) was dissolved in sulfuric acid and then mixed nitric and sulfuric acids added at a controlled rate to maintain relatively constant temperature (20°C.).

Following nitration the batch was gradually heated to 55°C. to complete the reaction.

Special precautions were taken to control heating because of known product instability above 150°C.

During the heatup cycle a violent reaction occurred, with considerable damage in all three floors of one 20' x 20' bay of a large manufacturing building. Three very minor injuries occurred.

The top head of the 500-gallon reactor was separated from the body of the vessel with enough force to throw it, accompanied by the agitator (a total of 540 lb.) a distance of over 500 feet.

Cause: A shortage of sulfuric acid shifted the sulfuric acid-intermediate ratio forming an unstable mixture on addition of nitric acid. When heated, an uncontrollable exothermic reaction occurred.

Calculations indicated decomposition could result in 2600 lb. pressure in the vessel from the volume of CO_2 released.

Thermal stability tests proved a serious exotherm at 60°C. resulting in development of 3300 lb. pressure.

Preventive Measures:

1. A better understanding is needed of potentially unsafe reactions especially those caused by an unbalance of reactants.
2. Positive means are being studied to assure the correct charge of critical components.

(REPORTED BY MANUFACTURING CHEMISTS' ASSOCIATION, INC.)

Reference Number of this Report: OI-110

Duplication of this Report is authorized

7/7/67

ARMED SERVICES EXPLOSIVES SAFETY BOARD
Nassif Building
Washington, D. C. 20315

OPERATIONAL INCIDENT REPORT NO. 111

Detonation in "Catch Box"

Description:

As part of a routine safety inspection, a technologist lifted the lid of a catch box to check the box and contents. While he was lowering the lid, an explosion occurred in the box, hurling the 38-lb. aluminum lid (28" x 54" x 1/4") into his face.

Cause:

It is believed that non-explosive intermediates combined with the contents (lead salts) of the catch box producing explosive material one of which was lead azide. The friction of closing the aluminum lid detonated some of these crystals.

Preventive Measures:

1. Catch boxes will be eliminated from laboratory installations.
2. All chemical wastes will be destroyed chemically before being ditched.

(REPORTED BY MANUFACTURING CHEMISTS' ASSOCIATION, INC.)

Reference Number of this Report: OI-111

Duplication of this report is authorized.

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ARMED SERVICES EXPLOSIVES SAFETY BOARD
Washington, D. C. 20315

OPERATIONAL INCIDENT REPORT NO. 111

Fire Involving Pyrotechnic Composition

DESCRIPTION:

Pyrotechnic composition (illuminant) was being consolidated into cannisters for 155mm projectiles utilizing a seven-station press. A permanent type vacuum collection system was installed.

At the direction of the Unit Foreman, the operation had been running for approximately 1-1/2 hours with the vacuum system inoperative. The stripper station operator, just beyond the first fire increment station, saw a flash in the vicinity of the first fire consolidation station. The operator yelled an alarm, "shut off the press", and evacuated the building.

The building was one-story, 44' x 67'. It was constructed of concrete and transite walls, concrete floor and a roof supported by wooden rafters and decking. Extent of loss; $475,000 - building, equipment, and contents.

There were no injuries.

CAUSES:

1. Exact Cause - Unknown.

2. Possible Cause: Friction due to metal-to-metal contact.

Reference Number of this Report: OI-111

Duplication of this report is authorized.

11/6/67

Incl # 1

ARMED SERVICES EXPLOSIVES SAFETY BOARD
Washington, D. C. 20315

OPERATIONAL INCIDENT REPORT NO. 112

Fluorolube - Aluminum Detonation Point

The following incident adds information to Potential Incident Report No. 38 which discussed information developed by test relative to the subject material.

INCIDENT: A technician fitted a 3/4" No. 16 aluminum bolt which had been lubricated with Fluorolube into a aluminum block 2" x 3" x 5". The bolt was screwed down normally with an 8" wrench for about 6 threads when it seized. Upon application of additional force a detonation resembling a 30 caliber rifle shot took place. The bolt was not ejected and the block was not cracked. The technician was not injured although black soot was sprayed on his arms. As both the block and bolt had been thoroughly cleaned it is highly unlikely that any contaminant was involved.

CONCLUSION: When Fluorolube is used as a lubricant with aluminum fittings, detonations may occur under conditions of galling and seizing.

RECOMMENDATION: Fluorolube, Kel-F and Halocarbon lubricants should not be used with aluminum.

Reference Number of this Report: OI-112

Duplication of this report is authorized.

11/6/67

Incl # 2

ARMED SERVICES EXPLOSIVES SAFETY BOARD
Washington, D. C. 20315

OPERATIONAL INCIDENT REPORT NO. 113

Ammonium Nitrate Explosion

DESCRIPTION: The accident occurred in a screw conveyor used to convey recycle and solid raw material to the granulator unit, as part of a process for the manufacture of a compound fertilizer from ammonium nitrate, ammonium phosphate and potassium chloride. This conveyor was constructed of mild steel and had a hollow shaft formed of 5-1/2 in. external diameter mild steel tube 3/8 in. thick and 12 ft. 3 in. long. This tube was machined to form a close fit over the solid shaft at the driving end and was open at the end which projected into the granulation unit. At the time of the accident, work was being carried out on the straightening of the blades forming the flights of the screw and and oxy-propane gas burner was being used for this purpose.

In the course of the work, an explosion occurred resulting in a rupture of the hollow steel shaft over a distance of about 3 ft. 6in. The conclusion of the investigation team who studied the circumstances of the accident was that over a period of about two years, the open end of the hollow shaft had admitted fertilizer compound material and in due course, the end had become completely sealed. The use of a gas torch on the repair work had caused the trapped material to decompose with resultant rise in pressure sufficient to disrupt the tube.

CAUSE: Decomposition of ammonium nitrate under high temperature and confinement conditions.

PREVENTIVE MEASURES:

1. Thorough inspection and decontamination of all locations where fertilizer material might become trapped prior to the use of any heat.

2. The elimination where possible of any hollow sections where fertilizer might enter.

Reference Number of this Report: OI-113

(REPORTED BY THE MANUFACTURING CHEMISTS ASSOCIATION)

Duplication of this Report is Authorized.

11/6/67

Incl # 3

ARMED SERVICES EXPLOSIVES SAFETY BOARD
Washington, D. C. 20315

OPERATIONAL INCIDENT REPORT NO. 114

Ammonium Nitrate Explosion

DESCRIPTION: The accident occurred with a closed hollow shaft of a screw conveyor; dimensions: length 7 m., diameter 250 mm., wall thickness 6 mm. This screw conveyor was used for transporting the raw materials and the recycle in the granulating plant. The shaft of this screw conveyor had a weak spot with a hole at one place, caused by scouring. The shaft was repaired by welding a bush around the weak spot with the hole. Two hours after this welding job, two men were working on this third floor of the building, lining the hopper of the screw conveyor with rubber sheets. At the moment that the shaft burst with explosive force, one man standing on top of the conveyor was killed instantaneously. The other man was thrown over the guard rail and landed on a floor 18 m. below. This man died in the hospital.

CAUSE: After investigation it was concluded that the hollow shaft was contaminated with NPK-type fertilizer through the hole at the worn spot. The nitrate containing fertilizer had been ignited by the heat from the welding. A self-sustained decomposition started after closing the hole and built up pressure until the shaft burst. As known, "cigar burning" is stimulated by pressure.

PREVENTIVE MEASURE: This serious accident shows that welding of hollow parts of granulating or fertilizer handling equipment could be very dangerous and precautions should be taken beforehand. Contaminated fertilizer in the hollow parts should be removed, e.g. by water, before welding.

Reference Number of this Report: OI-114

(REPORTED BY THE MANUFACTURING CHEMISTS ASSOCIATION)

Duplication of this Report is Authorized.

11/6/67

Incl # 4

ARMED SERVICES EXPLOSIVES SAFETY BOARD
WASHINGTON, D. C. 20315

OPERATIONAL INCIDENT REPORT NO. 115

Fire in Machining, Inspection and Inhibiting House

DESCRIPTION OF BUILDING:

The building was a single-story, wood frame structure approximately 58 feet by 116 feet. The roof was asbestos rolled roofing over wood sheeting on wood truss framing. The sub-floor was concrete with wood covered with asphalt tile. The interior finish was plywood.

DETAILS OF OCCURRENCE:

1. An operator in a propellant machining bay heard a noise and saw flame coming from an EXCELLO saw that was being used to cut grains of N-5 propellant to length.

2. Since the installed automatic sprinkler deluge system did not function, the operator pulled the chain attached to a manual trip valve and rushed from the bay. As he left the building, he also pulled a chain (outside the building) that was attached to another manual trip valve. The chain broke and the sprinkler deluge system was not activated.

3. During this time, the work leader observed the fire and rushed from the building, alerting other employees. He too noticed the failure of the automatic sprinkler deluge system to function and began to pull other manual trip valves located outside the building. After attempting (unsuccessfully) to activate the system, he went to another building and telephoned the fire department. Two fire trucks arrived at the scene by 1223 hours. The fire, which had propagated to adjacent propellant processing bays, was extinguished by 1245 hours.

NUMBER AND NATURE OF INJURIES: None

EXTENT OF LOSS: $24,812 building and $32,400 equipment and contents

CAUSES:

1. Exact cause: Unknown.

2. Possible cause: Ignition of propellant wafers by friction when caught between the saw and the saw cover.

REMARKS:

There was no automatic fire alarm connected to the fire department. Fire alarm boxes were not provided for reporting fires. The automatic sprinkler deluge system failed to function automatically and manually. The failure of the system allowed the fire to continue unchecked. The fire propagated to adjacent bays and to the roof of the building.

Reference Number of this Report: OI-115 (Duplication of this Report is Authorized)

12/1/67

ARMED SERVICES EXPLOSIVES SAFETY BOARD
WASHINGTON, D. C. 20315

OPERATIONAL INCIDENT REPORT NO. 116

Ignition of Igniter Pellets During Crimping Operation

DESCRIPTION:

Employee was operating a small bench type air press to crimp the rim of the metal igniter cup over a plastic top when ignition took place in a cup in the crimping fixture. It is believed that a pellet was caught between the rim of the cup and the cover and ignited from the impact of the crimping operation. Fire from the cup was directed to the rear of the press through vent holes in the crimping fixture. Some additional trays of charged cups were on the bench at the rear of the press and two trays from which the operator was working were between her and the press. To the left of the press was a feed chute leading to a hopper behind a reinforced concrete wall. The fire spread to the trays and through the chute to a can of black powder in the bay behind the reinforced concrete wall. The concussion created by the burning of the black powder blew out the roof and outside wall (blow out type) of the bay. Injury--minor burns on face and right hand (safety glasses protected eyes), and more serious burns on the left arm. Coveralls (flame proofed) were scorched from shoulders to waist but employee was not burned on this part of her body.

PREVENTIVE MEASURES:

1. Additional Shielding for the press and the trays awaiting assembly will be provided.

2. A flash tube extending from the crimping fixture directly to the outside will be installed.

(REPORTED BY THE MANUFACTURING CHEMISTS·ASSOCIATION)-Case History No.-1339

Reference Number of this Report: OI-116

Duplication of this Report is Authorized

12/1/67

ARMED SERVICES EXPLOSIVES SAFETY BOARD
WASHINGTON, D. C. 20315

OPERATIONAL INCIDENT REPORT NO. 117

LIQUID HYDROGEN SPILL DURING TRANSFER OPERATION

SUMMARY

During a routine transfer of liquid hydrogen from a commercial tank-trailer to a receiving vessel, a leak developed in the bayonet fitting at the trailer/facility connection. The leak produced a fan-shaped hydrogen vapor spray which enveloped the rear of the truck where the hand-operated shutoff valve was located. Emergency-trained personnel, wearing protective clothing, entered the area of the dense spray and successfully shut off the flow-control valve.

There was no damage to the facility or delivery systems. Members of the reentry party suffered minor frost-bite of their feet when their shoes became frozen to the water-wetted rear deck of the truck. The "freezing" of the shoes was caused by the extremely cold slush in which the men were standing.

ACTIONS TAKEN FOLLOWING LEAK INITIATION

Operating personnel actions, to control promptly all possible sources of ignition in the surrounding area, included shutdown of an opeating flare stack. Water hoses, which had been charged prior to the operations, were brought into play in an unsuccessful attempt to freeze over the leaking connection. With the leak developing into an uncontrollable situation, the operating personnel were faced with the choice of two alternatives: 1) to permit the leak to continue which, based on the estimated leak rate and the liquid hydrogen volume remaining in the tank, would have sustained the condition for up to 12 hours; or 2) to execute a reentry plan to close the tank shutoff valve at the rear of the truck. The latter course of action was chosen, and members of a reentry party, wearing protective clothing, were successful in closing the shutoff valve and terminating the leak.

CAUSE

A loose hose flange connection allowed leakage of cold fluid through the fluorocarbon lubricated bayonet seal. This leak allowed cold cryogenic fluid to contact and shrink the "O" ring seal, made of Buna-N synthetic rubber, thus permitting liquid hydrogen leakage to the atmosphere.

COMMENTS

This operating experience attests that:

1. Working in the midst of a cryogenic spill is a highly hazardous practice.

2. Use of a tank-trailer which does not have a safely accessible auxiliary shutoff valve is unsafe in the event of a spill.

Reference Number of this Report: OI-117

Duplication of this Report is Authorized

4/1/68

ARMED SERVICES EXPLOSIVES SAFETY BOARD
WASHINGTON, D. C. 20315

OPERATIONAL INCIDENT REPORT NO. 118

PNEUMATIC CYLINDER ROD UNSCREWS--DROPS DRYBOX DOOR

DESCRIPTION:

Two laboratory employees were working at a drybox, which was one of a number in series. One of the employees opened the air-operated vertical door on the drybox module and was about to pass some materials through. When the door reached the top of its travel and hit against the top of the door assembly housing, it immediately fell forcibly to the closed position. This guillotine action, if the door had fallen on a hand, wrist, or arm, would certainly have produced serious injury. Fortunately, no one was reaching under the door at the time.

The door is constructed of two $\frac{1}{4}$"-thick sheet steel panels. Between the two panels are a vertical rod and two horizontal rods which connect to the panels with spreader links. The vertical rod is fastened to the pneumatic cylinder (mounted outside on top of the door housing) piston rod by a threaded connection.

After disassembly and decontamination, it was found that the rod which fastens the piston to the door closing assembly had unscrewed, allowing the door to free fall. After consultation, it was determined that this inadvertent unscrewing of the piston rod from the door closing assembly could be prevented by drilling a small hole through the top of the door assembly lifting rod and the piston rod and inserting a cotter pin. This corrective measure will prevent rotation of the piston and rod in the pneumatic cylinder.

There are 63 of these air-operated doors installed in this laboratory. There is no way to examine these doors from the outside to see if this unscrewing has occurred, since the entire door assembly and lifting rod connection is enclosed inside the housing.

PREVENTIVE MEASURES:

It is recommended that laboratories having doors of this type examine and modify them as described above to eliminate this potentially serious hazard. Until this modification has been accomplished, personnel who are using air-operated drybox doors are cautioned to avoid reaching through them. Each door, when used, should be blocked with a stop to prevent its falling, or materials should be passed through with a carrier, so that no part of the body is placed under a door at any time.

Reference Number of this Report: OI-118

Duplication of this Report is Authorized 6/12/68

ARMED SERVICES EXPLOSIVES SAFETY BOARD
WASHINGTON, D. C. 20315

OPERATIONAL INCIDENT REPORT NO. 119

FLASH FIRE INVOLVING INITIATOR ASSEMBLIES FOR PRIMERS

DESCRIPTION:

Two employees, a truck driver and a production worker, were removing trays of initiator assemblies for primers from a van truck.

Eighteen trays of initiator assemblies were removed from a dryer, then loaded into a cargo van type truck for transportation to a production building. The vehicle proceeded to its destination. A production worker guided the truck driver in backing the vehicle to the loading platform. The production worker and truck driver then went to the rear of the truck. The production worker opened the doors of the truck. When he attempted to remove one of the trays, a flash fire occurred.

PREVENTIVE MEASURES:

A SOP should be developed for the packing, handling, blocking and staying of dangerous materials designated for intra-plant motor vehicle transportation. A review of work areas and job assignments should be made to determine where special personal protective clothing and equipment is required. Supervisors of explosives operations should be reinstructed regarding acceptance of tneir delegated safety responsibilities. Safety training of workers, the correction of unsafe acts or condition and supervisory enforcement of safety regulations should be emphasized.

Reference Number of this Report: OI-119

Duplication of this report is authorized

ARMED SERVICES EXPLOSIVES SAFETY BOARD
Washington, D. C. 20315

ADDITIONAL INFORMATION POTENTIAL INCIDENT RPT. NO. 38

Fluorolube - Aluminum Detonation Point

FLUOROCARBON LUBRICANTS AND ALUMINUM

It has been known for over 10 years that fluorocarbon greases and oils can explosively react with aluminum under shear loads. In 1960, a series of tests were conducted using drill tests, that is, a blunt end of a rotating drill with fluorocarbon oil is forcefully pulled down on a piece of aluminum. Other materials tested with aluminum included Kel-F oils and greases, Kel-F powders, and Teflon tapes and powders. No detonations were experienced with Teflons so it may be concluded that under most conditions Teflons would be safe to use. It was also found that explosions do not occur with all types of aluminum and there is evidently a need to determine which alloys produce unfavorable conditions.

At present there are three commercial products which react explosively with aluminum, they are; Fluorolube, Kel-F, and Halocarbon.

At present a new grease, Krytox, is being tested and apparently it will produce a detonation only under extremely severe conditions. It can be concluded that until the mechanism of these detonations is more fully understood no fluorocarbon lubricant should be used with either aluminum or magnesium.

FLUOROCARBON - ALUMINUM COMPATIBILITY

Fluorocarbon oils have been known to explode with aluminum under high shear loads and testing has been done in an attempt to clarify aluminum compatibility.

The laboratory reporting this testing reported that controlled explosions can be obtained by a drill press test. This is to say, a blunt end of a rotating drill having fluorocarbon oil on it, can cause an explosion when forcefully pulled down on a piece of aluminum. Although this test does not simulate service conditions, it is a test method which will select the materials that are more sensitive to shear reactions.

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Incl # 5 w/atch

A number of halogenated materials were tested in the above fashion. The results are shown in the attached table. It is interesting to note that all materials that detonated contain both fluorine and chlorine. It is reported that these materials are all polymers of chlorotrifluoroethylene, although they are marketed under various names. Kel-F, being one of the familiar names, was made to explode in the oil, powder and lip seal form. Teflon, which is polytetrafluoroethylene, a solid which contains no chlorine, could not be made to explode. In addition Aroclor 1254, a chlorinated biphenyl used as the liquid carrier in LOX SAFE, NA2-20502, did not explode. Based on these results it should be safe to use Teflon 30 for Naflex applications since the material should not be exposed to conditions with anywhere near the severity devised in the drill press test.

It is pointed out that the explosions do not occur with all types of aluminum. It isn't known why some of the aluminums do not detonate, but it does indicate that further work is warranted on this type of compatibility study to see if a condition does exist where the teflon could be detonated.

One theory is that the chlorine in the Kel-F material is the instigator of the explosion. Furthermore, the evidence presented here supports this theory. It is reported that a fully fluorinated grease can react with finely divided aluminum at a temperature of about 1200°F, but the same reaction with Kel-F oil occurs at about 425°F. This is explained by the fact that the stronger fluorine bond takes more energy for breakage than the chlorine bond. Moreover, the structure of the molecule apparently plays an important part in the shear reactions. The Aroclor 1254, a chlorinated carbon ring compound, is inert to aluminum in shear testing, yet the straight chained ethylene polymer which contains some chlorine does react.

Reference Number of this Additional Information Potential Incident: PI-38

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DRILL PRESS TEST
Using 3/4" Rivet Set at 500 RPM, and 950 psi Loading

Test No.	Type of Aluminum	Type of Halocarbon	Time of contact, Seconds	Type of Reaction	Remarks
1	6061-T6	Fluorolube LB	4.5	+	Faint detonation
2	"	"	2.2	+	Small flash
3	"	"	5.5	+	2 faint detonations 1 small flash
4	"	"	5.8	+	2 faint detonations
5	"	"	2.8	+	2 faint detonations
6	"	"	4.2	+	Flash
7	"	"	5.2	+	Flash
8	"	"	4.1	+	Faint Detonation
9	"	"	17.5	+	Small flash
10	"	"	4.8	+	Small flash faint detonation
11	6061-T6	Kel-F #90 Grease	12.1	+	Flash
12	"	"	22.0	+	Small flash
13	"	"	9.5	+	Small flash
14	"	"	6.2	+	Flash
15	"	"	9.7	+	Small flash
16	"	"	7.8	+	Flash
17	"	"	9.9	+	Flash loud detonation
18	"	"	5.5	+	3 small flashes 2 detonations
19	"	"	24.0	+	Smoke
20	"	"	8.7	+	Small flash
21	6061-T6	Kel-F #10 Oil	7.1	+	Faint detonation
22	"	"	8.2	+	Small flash
23	"	"	2.5	+	2 small flashes
24	"	"	1.9	+	2 small flashes
25	"	"	3.5	+	2 small flashes
26	"	"	30.0	-	---
27	"	'	21.0	+	2 small flashes
28	"	"	4.5	+	3 small flashes
29	"	"	2.8	+	3 small flashes
30	"	"	26.6	+	
31	6061-T6	Teflon #30 Dispersion	25	-	
32	"	"	25	-	
33	"	"	60	-	
34	"	"	40	-	
35	"	"	30	-	
36	"	"	60	-	
37	"	"	30	-	
38	"	"	30	-	
39	"	"	30	-	
40	"	"	30	-	

41	6061-T6	Kel-F Powder	3.6	+	Small flash
42	"	"	5.5	+	2 sm. flashes, blk residue
43	"	"	3.3	+	2 small flashes
44	"	"	30.0	-	
45	"	"	10.0	+	Small flash, blk residue
46	"	"	13.4	-	
47	"	"	30.0	-	
48	"	"	13.3	+	Smoke, and black residue
49	"	"	8.0	+	Small flash, blk residue
50	"	"	30.0	-	
51	6061-T6	Teflon Powder	30.0	-	
52	"	"	30.0	-	
53	"	"	30.0	-	
54	"	"	30.0	-	
55	6061-T6	Teflon Tape	10.2	-	
56	"	"	17.7	-	
57	6061-T6	Aroclor 1254	30.0	-	
58	"	"	30.C	-	
59	"	"	30.0	-	
60	"	"	30.0	-	
61	"	"	30.0	-	
62	"	"	30.0	-	
63	6061-T6	Halocarbon 25-20MZ	12.3	+	Flash, & detonation
64	6061-T6	Florube A	30.0	-	
65	"	"	2.5	+	Small detonation
66	2024 T4	Fluorolube LG	30.0	-	
67	"	"	30.0	-	
68	"	Fluorolube MG	30.0	-	
69	"	"	30.0	-	
70	"	Halocarbon 25-20 25-20 MZ	30.0		
71	6061-T6	Fluorolube LG	2.5	+	Flash (470 psi load)
72	"	"	4.3	+	Flash (470 psi load)
73	"	"	3.2	+	Flash (90 psi load)
74	6061-T6	Kel-F Lip Seal	*135 *Includes time to wear thru seal	+	Flash & detonation (470 psi load)
75	2024-T4	Fluorolube LG	210	-	

76	7075-T6	Fluorolube LG	43	-	
77	"	"	20	+	Flash & detonation
78	"	"	14.3	+	Flash & detonation
79	"	"	9.5	+	Flash & detonation (Alclad sanded)
80	5052-0	Fluorolube LG	60	-	
81	5052-0	"	3.7	+	Flash and detonation
82	"	"	30	-	
83	"	"	30	-	
84	"	"	6	+	Small detonation
85	5052-H34	Fluorolube LG	30	-	
86	"	"	29	+	Small detonation
87	"	"	54	+	Very small detonation
88	"	"	4.8	+	2 very small detonations
89	3003-0	Fluorolube LG	5.9	+	Flash and detonation
90	"	"	5.3	+	Flash and detonation (470 psi)
91	Pure Aluminum	Fluorolube LG	100	-	
92	"	"	120	-	
93	13% Silicon Aluminum	Fluorolube LG	4	+	Flash and detonation
94	AN Fitting	Fluorolube LG	60	-	
95	"	"	120	-	
96	2014	Fluorolube LG	7.5	-	
97	"	"	2.5	+	Flash and detonation
98	"	"	4.3	+	Flash and detonation
99	Tens-50	Fluorolube LG	-	-	Set walked off specimen
100	"	"	30.0	-	
101	"	"	30.0	-	

ARMED SERVICES EXPLOSIVES SAFETY BOARD
WASHINGTON, D. C. 20315

Potential Incident Report No. 39

Very little information has been written in the general chemical field on Sensitivity of Metal-Halogenated Solvent Combinations. The inclosed paper is a summary of certain investigations on the subject and reveal potential hazards.

Reference Number of this report: PI-39 - This report may be duplicated.

1/8/68

SENSITIVITY OF METAL-HALOGENATED SOLVENT COMBINATIONS

by

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A Division of North American Rockwell Corporation

1/8/68

SENSITIVITY OF METAL-HALOGENATED SOLVENT COMBINATIONS

Impact sensitivity tests were found to be beneficial in determining the sensitivity of barium/Freon TF combinations. Because of the widespread use of halocarbons and potentially reactive metals within the industry, it was deemed prudent to extend the impact sensitivity experiments to include a series of metals and halocarbons currently in use.

Literature surveys disclosed that very little has been written in the general chemical field on barium and its reactivity. Only when the literature surveys include the pyrotechnics and explosives areas do we begin to find indications of the explosive reactivity of barium and halocarbons. This same literature survey also disclosed that granular metals other than barium have on occasion reacted unexpectedly and explosively with halogenated solvents, including aluminum powder with carbon tetrachloride and aluminum powder with trichloroethylene. This type of behavior of powdered or granular metals after exposure to supposedly "safe" solvents raises considerable doubt about their safeness. The program described herein was conducted to determine just how sensitive to impact various metal/halocarbon slurries are, and to determine, if possible, if a potential hazard (due to impact) exists in the handling of more common powdered or granular metals in contact with a variety of commonly used halogenated solvents. The combinations chosen for this program are representative of the kinds that are in general use; they include some combinations known to be or likely to be used in manufacturing or laboratory operations. Ten different kinds of metallic powders or granules were tested with six different solvents, giving a total of sixty combinations. A brief description of these particular metallic materials and solvents is as follows:

METALS:

Aluminum Powder	- Reynolds No. 1-511 Atomized Powder, 13 ± 3 microns, Reynolds Metals Company
Mg Powder	- 30/50 Mesh Atomized, Harshaw Chemical Corp.
TI Powder	- Laboratory sample labeled " +325 mesh", origin unknown.
Ba Shavings	- Shavings produced by cutting up nominal 1 mm X3 mm granules, Alfa Inorganics, Inc.
Li Shavings	- Small shavings cut from an ingot, Lithium Corp. of America.
Be Powder	- Fine Powder (size unknown) Brush Beryllium Corp.
BeH_2 Powder	- Fine Powder (size unknown) Ethyl Corp
Aluminum Filings	- Filings produced with a coarse rasp from a sheet of commercial aluminum (grade unknown).
Magnesium Filings	- Filings produced with a coarse rasp from a sheet of commercial magnesium (grade unknown).
Boron Powder	- 95 +% Purity, average particle size less than 1 micron, American Potash and Chemical Corp.

SOLVENTS:

Freon MF CCl_3F	-	Monofluorotrichloro methane, E. I. duPont
Freon TF $C_2Cl_3F_3$	-	Trichlorotrifluoro ethane, E. I. duPont
Carbon Tetrachloride CCl_4	-	Mallinkrodt Chemical Works, Analytical Reagent (low Sulfur)
Trichloroethylene C_2HCl_3	-	Mallinkrodt Chemical Works, Analytical Reagent
Perchloroethylene C_2Cl_4	-	Tetrachloroethylene, Matheson Coleman and Bell, Spectroquality Reagent
1,1,1 Trichloroethane CH_3CCl_3	-	Methyl Chloroform, Matheson Coleman and Bell, Technical Grade

The impact sensitivity test apparatus used on this program is a slight modification of the Jet Propulsion Laboratory (JPL) impact sensitivity tester. Basically, the test is conducted by putting a small sample (a few milligrams) of the material to be tested inside a shallow depression in a hardened steel anvil, then dropping a steel ball of a known weight from a measured height so that it strikes a hammer which is in contact with the sample. A "go" on this tester is evidenced by either a flash or a loud noise or both. By increasing the drop height after each "no go" or lowering the height after a "go" it is possible to get some idea of the impact sensitivity of the material being tested. The sample is changed after each "no go" so that a fresh sample is used for every test.

Most of the many kinds of testers in current use appear to give a different impact sensitivity value for the same material. For this reason it is generally necessary to reference the results obtained for a new material against some well-characterized material tested on the same impact machine. This type of comparison is more meaningful than an absolute value stated in some units of weight x height would be, even though these absolute values are often quoted (for a given type of tester) as an indication of the impact sensitivity of a material. The reference material used in this program was Composition B, a high explosive consisting of a blend of RDX and TNT with a small amount of wax added.

Most of the metal-solvent combinations tested were run in an identical manner. A quantity (not weighed) of the powdered or granular sample sufficient to fill the cavity in the anvil was used in each test. When the solvent was added in excess and allowed to remain in contact with the powder for about a minute prior to dropping the ball. Where solvent had a tendency to evaporate, especially evident with Freon MF, additional solvent was added at intervals so that an excess was present at all times up to the time of impact.

With test involving barium it was believed desirable because of the reactivity of barium with oxygen and moisture to minimize or eliminate exposure to air prior to the impact of the ball*. Accordingly, whenever barium was handled it was always done in an argon atmosphere up until the sample could be "drowned" in the solvent. Lithium, also reactive, was cut into shavings under an argon atmosphere but less care was taken to exclude air in subsequent operations. An attempt was made to minimize exposure to air of the aluminum filings and the magnesium filings by producing only enough filings at one time for a few tests, then testing them very quickly afterward. In all the other tests no attempt was made to exclude air from the metals at any time.

The maximum drop height capability of the test apparatus employed is 50 inches. With a 5-pound ball, any material that fails to show any reaction at 50 inches is usually considered to be relatively insensitive to initiation by impact. (Composition B, previously mentioned as our reference material, showed a 50% probability of detonating at 8 inches on our tester.)

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*There is some evidence that moderate exposure of barium to air does not materially affect its sensitivity. Prior to running this present series of 60 combinations, it had been found in an earlier test that the barium-Freon TF combination in which no attempt was made to exclude air after the barium had been cut into shavings gave flashing at a 3-inch height with a 5-pound ball. This compares very closely with the value of 4 inches with the same 5-pound ball, found during the current tests where air exposure was eliminated. The difference between the 3-inch height and 4-inch height is considered to be of little significance, probably well within the experimental range of values to be expected from this type of test.

Ordinarily the impact sensitivity heights reported in the literature are 50% probability levels, although in some instances the height given is the 10% probability point. Both levels are arrived at by some up or down system (normally Bruceton series) of varying the height from test to test and require that fairly large numbers of tests be run. In the current program the objective was more concerned with determining the approximate minimum height at which a reaction occurred, rather than determining the statistical probability of this event occurring. Because of the limited number of tests run on each combination, it is reasonably certain that the minimum heights given for many of the combinations in Table I are higher than would have been obtained had a larger number of samples been tested.

The maximum height of 50 inches was utilized for the initial testing of most combinations. If three consecutive "no go's" resulted, it was presumed that this particular combination was insensitive to impact and no further tests were run on this combination. If any of these initial three tests produced a detonation or flash, the height was progressively reduced until a height was reached where three consecutive tests failed to give a go, at which point testing of that combination was stopped. Therefore the values given in Table I are these heights at which a reaction occurred at least once out of three attempts.

Looking at Table I, a number of things are evident. Probably the most evident is that combinations with barium are definitely the most sensitive of all combinations tried. This sensitivity, greater even than lithium combinations, is at first glance very surprising, since lithium is a member of the extremely reactive alkali metal series, and is higher in the electromotive series than barium. However, the German references also showed the barium slurries to be both more sensitive to initiation and more violent in their reaction than lithium slurries with the same halocarbon. The value of 4 inches at which a flash occurred (as mentioned previously, an earlier series of tests had given a value of 3 inches) indicates that the barium-Freon TF combination is a very hazardous one. In fact, Freon TF combinations were among the most reactive with all the metals tested.

The term "detonation" as used here indicates that a loud noise, usually accompanied by a bright flash and smoke, were produced. There was also a strong pungent odor produced, and the test apparatus and immediate vicinity were covered with a film of gray or black material. None of the original sample was found in the apparatus.

Where only flash or heavy sparking was recorded, the only noise that could be detected was the background noise produced by the impact of the ball striking the test apparatus. In these instances part of the original test sample was found to be still in the apparatus, just as it was when there was no evidence of a reaction.

Table I shows that one of the metals gave a positive reaction with 1,1,1 trichlorethane (methyl chloroform). This might indicate that this would be a safe solvent to use. Unfortunately, this conclusion is not necessarily a valid one. Although the tests show that 1,1,1 trichloroethane is safer, its composition is very similar to some of the other solvents, so that under the proper conditions it might react much as these other solvents do.

Based on the results of this program and on the limited, brief literature search, there is a definite potential hazard in handling granulated metal-halogenated solvent combinations. Serious consideration should be given to the use of nonhalogenated solvents whenever possible, even if it means increasing the flamability hazards of the operation. Adequate measures (ventilation, inert atmospheres, grounding, etc.) can usually be taken to reduce this flammability hazard.

The use of an ever-increasing variety of new metals and alloys in industry may produce some very sensitive combinations if these materials are exposed to halogenated solvents. Before any halogenated solvents are used with powders, shavings, chips, etc, of these new metals or alloys the reactivity should be determined by some sort of impact sensitivity tests as a minimum requirement. It might be wise to run other sensitivity tests as well, since a combination which appears to be insensitive to impact may be sensitive to another mode of initiation.

These precautionary measures with halocarbons are probably not necessary when handling large pieces of sheet metal, billets, castings, or machined parts, <u>if</u> proper care is taken to previously ensure the removal of chips, cuttings, filings, and grindings from the parent material.

TABLE I

MINIMUM IMPACT SENSITIVITY HEIGHT (inches)

(5-lb Ball)

SOLVENT	METAL									
	Al Powder	Mg Powder	Ti Powder	Ba Shavings	Li Shavings	Be Powder	BeH_2 Powder	Al Filings	Mg Filings	B Powder
Freon MF	F,50	0	0	X,20 F,15	X,50 F,40	0	0	0	0	0
Freon TF	F,50	0	F,50	X,10 F,4	X,20	0	0	0	0	0
Carbon Tetrachloride	X,50	F,50	0	X,15 F,10	X,18	F,50	0	0	0	0
Trichloroethylene	0	F,50	F,50	X,15 F,13	X,25	F,50	0	0	0	0
Perchloroethylene	0	0	0	X,20 F,15	X,30	0	0	0	0	0
1,1,1 Trichloroethane	0	0	0	0	0	0	0	0	0	0

X,Number - Height in inches at which detonation occurred.

0 - No reaction at 50 inches.

F,Number - Height in inches at which flash or heavy sparking occurred